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ROUTINE INSTRUMENT SETTINGS FOR VOLATILES ANALYSIS

FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION

CHEMISTRY PROGRAM

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ROUTINE INSTRUMENT SETTINGS FOR VOLATILES ANALYSIS

1. SCOPE AND APPLICATION

1.1. The settings for analytical instrumentation used within the FDEP volatiles (VOC) laboratory are provided in this Standard Operating Procedure (SOP). The values are guidance and may vary somewhat from instrument to instrument in order to optimize performance. Once established, the settings must not be altered for a particular instrument calibration. If the settings are altered or the equipment is serviced, then a new calibration must be prepared.

1.2. FDEP Test IDs and equivalent EPA methods covered by this SOP:

Test	Matrix	EPA Method Reference and Description	Instrument Set-up
W-VOC-MS	Water	Method 624.1 analysis, non-preserved	Sections 2.1.1. and 2.2.2.
W-VOC-MS-A	Water	Method 624.1 analysis, acid-preserved	Sections 2.1.1. and 2.2.2.
W-VOC-MS-R	Water	Method 8260D analysis, non-preserved	Sections 2.1.1. and 2.2.2.
W-VOC-MS-R-A	Water	Method 8260D analysis, acid-preserved	Sections 2.1.1. and 2.2.2.
S-VOC-MS	Soil	Method 8260D analysis, Sediment	Sections 2.1.2. and 2.2.2.
WAS-VOC-MS	Waste	Method 8260D analysis, Waste	Sections 2.1.1. and 2.2.2.
TCLP-VOC	Waste	Method 8260D analysis of TCLP Extract	Sections 2.1.1. and 2.2.2.
SPLP-VOC	Waste	Method 8260D analysis of SPLP Extract	Sections 2.1.1. and 2.2.2.
T-VOC-MS	Tissue	Method 8260D analysis, Tissue	Sections 2.1.3. and 2.2.2.
W-8015	Water	Method 8015C analysis, acid preserved	Sections 2.1.4. and 2.2.2.
W-624-AA	Water	Method 8260D analysis, non-preserved	Sections 2.1.4. and 2.2.2.
W-ETHYL-AC	Water	Method 8260D analysis, acid-preserved	Sections 2.1.4. and 2.2.2.
W-FUMGNT-A	Water	Method 8260D analysis, acid-preserved	Sections 2.1.5. and 2.2.3.
W-1-4-DIOX	Water	Method 522 analysis, Na2SO3/NaHSO4	Section 2.2.4.
W-FUM	Water	Method 8260D analysis, non-acid-preserved	Sections 2.1.5. and 2.2.3.

1.3. Refer to the specific analytical method SOPs for details regarding sample preparation and analysis, apparatus/equipment, and reagents/chemicals.

SOP **VO-002** Analysis of VOCs in Water by EPA Method 624.1/8260D
SOP **VO-003** Analysis of VOCs in Sediment, Tissue, and Waste by EPA 8260D
SOP **VO-004** Analysis of Polar Organic Compounds in Sediment and Waste
SOP **VO-005** TCLP and SPLP Sample Preparation
SOP **VO-006** Analysis of Polar Organic Compounds in Water
SOP **SV-015** Analysis of 1,4-Dioxane in Water by EPA Method 522

2. VOC INSTRUMENT SETTINGS

The following settings are for an Agilent 7890 Gas Chromatograph (GC) and 5975C (or 5977) Inert Mass Spectrometer (MSD) equipped with a Tekmar Atomx Purge & Trap System (or similar equipment).

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The operation, cleaning, and scheduled maintenance procedures prescribed by the equipment manufacture are followed as provided in the Operator's Manuals. Maintenance contracts, provided by the manufacture, are purchased for the Gas Chromatograph/Mass Spectrometer Systems. Documentation of cleaning, maintenance, or system modifications is recorded in a maintenance logbook which accompanies each instrument system.

2.1. Purge & Trap Setting - Tekmar Atomx

2.1.1. EPA Methods 624.1/8260D Water Method

• Helium Purge Flow Rate: 38 mL/min	• Drypurge Time = 1 min
• Trap Type: Trap #9	• Drypurge Temp = 20ºC
• Sample Volume: 15 mL	• Drypurge Flow = 100 mL/min.
• Sample Heater = On	• GC Start = Start of Desorb
• Sample Sparger Temp = 35ºC	• Desorb Preheat = 245ºC
• Purge Time = 11 min.	• Desorb Time = 1.0 min.
• Transfer Line Temp = 125ºC	• Desorb Temp = 250ºC
• Valve Oven Temp = 125ºC	• Bake Flow = 200 mL/min.
• Purge Ready Temp = 40ºC	• Sample Drain = On
• Purge Temp = 20ºC	• Bake Time = 5 min.
• Sample Mount Temp = 90ºC	• Bake Temp = 260ºC
• Condenser Ready Temp= 40ºC or 45ºC	• Condenser Bake Temp = 200ºC
• Condenser Purge Temp = 20ºC	• Standby Flow = 10 mL/min.

2.1.2. EPA Method 8260D Sediment Heated-Purge Method

• Helium Purge Flow Rate: 40 mL/min	• Soil Vial Mix Speed: Fast
• Trap Type: VOCARB 3000 or Trap #9	• Presweep Time = 0.25 min
• Water Volume: 5 mL or 10 mL	• Sweep Water Time = 0.25 min
• Sparger Sample Heater = Off	• Sweep Water Flow = 100 mL/min
• Soil Valve Temp = 100ºC	• Drypurge Time = 2.0 min
• Sample Vial Temp = 40ºC	• Drypurge Temp = 20ºC
• Sample Preheat Time = 0.0 min.	• Drypurge Flow = 100 mL/min.
• Transfer Line Temp = 125ºC	• GC Start = Start of Desorb
• Valve Oven Temp = 125ºC	• Desorb Preheat = 245ºC
• Purge Ready Temp = 40ºC	• Desorb Time = 2.0 min.
• Purge Temp =20ºC	• Desorb Temp = 250ºC
• Sample Mount Temp = 90ºC	• Standby Flow = 10 mL/min.
• Condenser Ready Temp = 45ºC	• Sample Drain = On
• Condenser Purge Temp = 20ºC	• Bake Time = 2 min.
• Soil Valve Temp = 100ºC	• Bake Temp = 260ºC
• Purge time = 12 min.	• Condenser Bake Temp = 200ºC
• Purge Flow = 40 mL/min	• Bake Flow = 300 mL/min
• Water Heater Temp = 90ºC	• Drain Flow = 300 mL/min

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2.1.3. EPA Method 8260D Tissue Heated-Purge Method

The tissue heated-purge settings were established using a Tekmar Atomx instrument that incorporates both autosampler and Purge&Trap functions into one sample introduction system.

• Helium Purge Flow Rate: 38 mL/min	• Presweep Time = 0.25 min
• Trap Type: Trap#9	• Sweep Water Time = 0.25 min
• Water Volume: 5 mL	• Sweep Water Flow = 100 mL/min
• Sparger Sample Heater = On	• Drypurge Time = 3.0 min
• Sparger Temp = 40ºC	• Drypurge Temp = 20ºC
• Sample Vial Temp = 55ºC	• Drypurge Flow = 100 mL/min.
• Sample Preheat Time = 1.0 min.	• GC Start = Start of Desorb
• Line Temp = 130ºC	• Desorb Preheat = 245ºC
• Valve Temp = 130ºC	• Desorb Time = 2.0 min.
• Purge Ready Temp = 40ºC	• Desorb Temp = 250ºC
• Purge Temp =20ºC	• Desorb Flow = 100 mL/min.
• Sample Mount Temp = 90ºC	• Sample Drain = On
• Condenser Ready Temp = 45ºC	• Bake Time = 10 min.
• Condenser Purge Temp = 20ºC	• Bake Temp = 260ºC
• Purge time = 12 min.	• Condenser Bake Temp = 240ºC
• Purge Flow = 40 mL/min	• Bake Flow = 300 mL/min
• Water Heater Temp = 90ºC	• Drain Flow = 300 mL/min
• Soil Valve Temp = 105ºC	• Standby Flow = 10 mL/min.
• Soil Vial Mix Speed: Fast	

2.1.4. EPA Methods 8260D/8015C Water Heated-Purge Method (* trap and temp options)

• Helium Purge Flow Rate: 40 mL/min	• Drypurge Time = 1 min
• Trap: Trap#9	• Drypurge Temp = 20ºC
• Sample Volume: 15 mL	• Drypurge Flow = 100 mL/min.
• Sample Heater = On	• GC Start = Start of Desorb
• Sample Sparger Temp = 63ºC	• Desorb Preheat = 245ºC or 210ºC *
• Purge Time = 11 min.	• Desorb Time = 1.0 min.
• Transfer Line Temp = 125ºC	• Desorb Temp = 250ºC or 220ºC *
• Valve Oven Temp = 125ºC	• Standby Flow = 10 mL/min.
• Purge Ready Temp = 40ºC	• Sample Drain = On
• Purge Temp = 20ºC	• Bake Time = 5 min.
• Sample Mount Temp = 90ºC	• Bake Temp = 260ºC or 220ºC *
• Condenser Line Temp = 45ºC	• Condenser Bake Temp = 200ºC
• Condenser Des Temp = 20ºC	•

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2.1.5. EPA Method 8260D Selected Ion Monitoring (SIM) Fumigant Water Method

• Helium Purge Flow Rate: 38 mL/min	• Drypurge Time = 2 min
• Trap Type: #1A Tenax	• Drypurge Temp = 20ºC
• Sample Volume: 20 mL	• Drypurge Flow = 100 mL/min.
• Sample Heater = On	• GC Start = Start of Desorb
• Sample Sparger Temp = 60ºC	• Desorb Preheat = 220ºC
• Purge Time = 11 min.	• Desorb Time = 2.0 min.
• Transfer Line Temp = 125ºC	• Desorb Temp = 225ºC
• Valve Oven Temp = 125ºC	• Standby Flow = 10 mL/min.
• Purge Ready Temp = 40ºC	• Sample Drain = On
• Purge Temp = 20ºC	• Bake Time = 5 min.
• Sample Mount Temp = 90ºC	• Bake Temp = 230ºC
• Condenser Ready Temp = 45ºC	• Condenser Bake Temp = 200ºC
• Condenser Purge Temp = 20ºC	•

2.2. Agilent 7890/5975 Gas Chromatograph/Mass Spectrometer (or equivalent) Settings

2.2.1. Analytical Column.: The Restek Rxi-624Sil MS, 30m x 0.25mm ID x 1. µm film thickness column (cat# 13868) is used for most volatile applications. This column has proven superior to the older J&W DB-624 columns used previously, but is likely equivalent to the DB-624 MS version. The Restek Rtx-VMS, 30m x 0.25mm ID x 1. µm column (cat# 19915) is used for the 1,4-dioxane and fumigant selected-ion monitoring tests. The Rtx-VMS column performs superior for the 1,4-dioxane analysis.

2.2.2. EPA Methods 624.1/8260D GC/MS (Full-Scan) Water and Sediment Program Settings

The GC/MS program provided below is used for most routine VOC water, tissue, and sediment analysis. The exceptions are the specialty SIM methods for fumigants and 1,4-dioxane.

GC Initial Temperature = 35ºC	GC Injector Temperature = 150ºC
GC Initial Time = 3 min.	GC Ramp 2 Rate =19ºC/min
GC Ramp 1 Rate = 6ºC/min.	GC Ramp 2 Final Temp = 220ºC
GC Ramp 1 Final Temp = 100ºC	GC Ramp 2 Final Time = 0 or 0.50 min.
GC Ramp 1 Final Time = 0 min.	GC Post Run =35ºC
GC Carrier Gas Flow Rate = 1.2 ml/min	GC Post Run Flow = 1.0 mL/min
GC Split Mode Ratio = 30:1	GC Gas Saver On at 20 mL/min
MSD Quad Temp = 150ºC	MSD Filament Delay = 1 min
MSD Source Temp = 230 ºC	MSD Acquisition Time = 20 min
MSD Transfer Line Temp = 220ºC	MSD Scan Range: 40-260 amu

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MSD Scan Mode = EI	MSD Scan Rate: 3.22, 4.32,4.45 6.1scans/s
MSD Acquisition Mode = Full Scan	

2.2.3. EPA Method 8260D GC/MS (SIM) Program Settings for Fumigants

GC Initial Temperature = 35ºC	GC Injector Temperature = 125ºC
GC Initial Time = 3 min.	GC Ramp 2 Rate =15ºC/min
GC Ramp 1 Rate = 5ºC/min.	GC Ramp 2 Final Temp = 200ºC
GC Ramp 1 Final Temp = 70ºC	GC Ramp 2 Final Time = 0 min.
GC Ramp 1 Final Time = 0 min.	GC Post Run =70ºC
GC Carrier Gas Flow Rate = 1.2 ml/min	GC Post Run Flow = 1.0 mL/min
GC Split Mode Ratio = 30:1	GC Gas Saver On at 20 mL/min
MSD Quad Temp = 150ºC	MSD Filament Delay = 1 min
MSD Source Temp = 230ºC	MSD Acquisition Time = 18.6 min
MSD Transfer Line Temp = 125ºC	MSD Scan Range: SIM ions
MSD Scan Mode = EI	MSD Scan Rate: 6.0 scans/sec
MSD Acquisition Mode = SIM	MSD Cycles/sec 1.38 to 4.6

2.2.4. EPA Method 522 GC/MS (SIM) Program Settings for 1,4 Dioxane

GC Initial Temperature = 35ºC	GC Splitless Mode
GC Hold Time = 3 min.	GC Injector Temperature = 200ºC
GC Ramp 1 Rate = 5ºC/min.	GC Post Run =70ºC
GC Ramp 1 Final Temp = 70ºC	GC Post Run Flow = 1.0 mL/min
GC Ramp 1 Final Time = 0 min.	GC Injection Volume = 1 uL
GC Carrier Gas Flow Rate = 1.0 ml/min	
MSD Quad Temp = 150ºC	MSD Filament Delay = 7 min
MSD Source Temp = 230ºC	MSD Acquisition Time = 15 min
MSD Transfer Line Temp = 125ºC	MSD Scan Range: SIM ions
MSD Scan Mode = EI	MSD Scan Rate: 3.3 scans/sec
MSD Acquisition Mode = SIM	MSD Cycles/sec 3.1-2.25

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